

# **A Comparative Study of the Quantitative Analysis of Polyester/Cotton Blends by AATCC Method 20A ("Fiber Analysis: Quantitative") and Differential Scanning Calorimetry (DSC)**

Le Tiet Hecking, US Customs Research Laboratory  
Textile Team, New York Laboratory

## **Introduction**

Note 2A, Section XI of the Explanatory Notes of the Harmonized Tariff Schedule of the United States (HTSUS), states that articles covered in Chapters 50 to 55, which are composed of two or more textile fibers are classified according to the component fiber of chief weight. A compositional determination is necessary, therefore, for the proper tariff classification of these goods. The Customs laboratories follow AATCC Method 20A, "Fiber Analysis: Quantitative,"<sup>1</sup> to analyze the various textile products containing natural/man-made or man-made/synthetic fibers. However, this procedure is time-consuming and requires the use of solvents. A more rapid testing procedure is needed for screening. Differential scanning calorimetry (DSC), used for the characterization of polymers, is investigated in this study because it is a fast and well-established technique, which requires neither the use of solvent nor a lengthy sample preparation.

This study was conducted to compare the DSC results to those obtained by AATCC Method 20A (chemical separation) in the quantification of polyester/cotton blends. The DSC procedure is described in this paper.

## **Experimental Procedures**

### **I. Samples**

#### **A. Fabrics**

The fabrics analyzed were imported textile fabrics submitted to a U.S. Customs field laboratory for analysis to determine the percent fiber composition for proper tariff classification. All fabrics used in this study were converted (e.g., no greige fabrics were examined), and included both woven and knitted fabrics. The yarns in these samples are intimate fiber blends that cannot be separated mechanically. Fabric specimens were analyzed whole; none of the samples were deconstructed (deknit) to component yarns. This technique may also be applied to analysis of fiber-blended yarns.

#### **B. Nonfibrous Materials**

General Explanatory Note (I)(A)(5) to Section XI of the HTSUS stipulates that dressings, sizings, and the like, are to be accounted for when reporting the weights of textile fibers (i.e., "... in the state in which they are presented ..."). Any such finishes must be removed prior to analysis, their weights calculated and reported. No size removal was undertaken either by the field laboratory in its chemical separation analysis, nor by the Research Laboratory in this preliminary DSC study. Further, none of the sample fabrics analyzed in this study exhibited any finishes visible to the unaided eye, such as coatings or impregnations of the kind addressed in Chapter 59 of the HTSUS.

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<sup>1</sup> 1998 *Technical Manual of American Association of Textile Chemists and Colorists*. AATCC Test Method 20 A-1995—"Fiber Analysis: Quantitative." AATCC. Research Triangle Park, NC. 1997.

## II. Procedures

### A. Chemical Separation (AATCC Method 20A)

Samples were analyzed “as received,” following AATCC Method 20A (chemical separation procedure). Details of the chemical separation analyses are not discussed in this paper. The data obtained by the field laboratory analysts are listed in Table 1 for comparison with those generated by the DSC technique.

### B. Differential Scanning Calorimetry (DSC)

The Research Laboratory analysis used a DSC 2920 (TA Instruments, Inc., New Castle, DE) equipped with an autosampler and a refrigerated cooling system. Nitrogen was used as purge gas, and compressed air for cooling.

Prior to scanning the samples, a two-point calibration<sup>2</sup> was performed using indium and lead standards from traceable sources, at the same heating rate as the samples to cover the melting range of polyester. The baseline calibration (cell calibration) was also performed using the same program as for the samples.

The samples were stored in the textile conditioning room [20°C (± 1°C) temperature, and 65% (± 2%) relative humidity] prior to sampling. Actual sample preparation, however, was completed outside of the textile conditioning room. All samples were analyzed “as received.”

Small fabric pieces, approximately 4 mm x 4 mm, were cut from the sample, and placed in the bottom of a tared non-hermetic aluminum sample pan. The sample pan was crimped using an appropriate TA Instruments, Inc. sample press. Each test specimen was weighed (5-6 mg) to an accuracy of 0.0001 mg, and each sample was analyzed in duplicate. All samples were subjected to the same heating/cooling/heating cycle.

The DSC was programmed to heat the test specimens at 10°C per minute from 35 to 300°C, and the temperature of 300°C was held for two minutes. The first heating melts the polyester and removes all artifacts associated with the manufacturing process. Cooling to 35°C allows the polyester to resolidify and to give it a known thermal history. After a hold for two minutes at 35°C, the specimen was heated again to 300°C to obtain the heat of fusion of the material with a known thermal history. A thermal curve was obtained. The curve of the second heating was integrated to obtain the heat of fusion, or enthalpy, “Delta H,” expressed in Joules/gram, which is used for the calculation of the percent composition.

Samples of 100% polyester fabric were scanned first to obtain an average of the heat of fusion, Delta H, of polyester (Delta H<sub>PET</sub>). The thermal curves of 100% polyester are shown in Figures 1 and 2. Then polyester/cotton-blended samples were scanned. Figures 3 and 4 show the thermal curves of a polyester/cotton sample. The percent composition of polyester in the sample was calculated using the following formula:

$$\% \text{ Polyester} = (\text{Delta H}_{\text{sample}} / \text{Delta H}_{\text{PET}}) \times 100$$

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<sup>2</sup> 1998 *Annual Book of ASTM Standards* (Vol. 08.02). ASTM D3417-97—“Standard Test Method for Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry (DSC).” ASTM. Philadelphia, PA. 1998.

The average of the Delta H<sub>PET</sub> obtained was 39.08 Joules/gram. Anecdotal a Delta H<sub>PET</sub> value of 39.0 Joules/gram has been reported in the context of a seminar,<sup>3</sup> then reconfirmed in private electronic communication (E-mail).<sup>4</sup> While that value tends to support those determined by this research, no single value should be considered as definitively representative of other polyesters.

## Results and Discussion

**Table 1. Data obtained by chemical separation and DSC analyses.**

SAMPLE ID	STATED COMPOSITION		CHEMICAL SEPARATION RESULTS		DSC RESULTS	
	POLYESTER	COTTON	POLYESTER	COTTON	POLYESTER	COTTON
10341	55.00	45.00	66.40	33.60	73.98	26.02
10383	65.00	35.00	60.20	39.80	56.17	43.83
10500	52.00	48.00	53.70	46.30	54.79	45.21
10550	50.00	50.00	45.90	54.10	47.29	52.71
11351	72.00	28.00	73.20	26.80	75.54	24.46
10698	40.00	60.00	46.30	53.70	41.89	58.11
10027	48.00	52.00	45.10	54.90	48.91	51.09
10028	65.00	35.00	77.60	22.40	78.28	21.72

As shown in Table 1, the DSC data are in agreement with those obtained by AATCC Method 20A. Where the chemical separation test determined a component of chief weight, DSC results concurred. In some instances, the results differed in assay; however, there is no change in the classification as a result of these differences.

A reproducibility study was also conducted using three samples fabrics containing 100% polyester, 14/86 polyester/cotton, and 75/25 polyester/cotton. Each test specimen was scanned on the DSC from 35 to 300°C at 10°C per minute, then cooled to room temperature. Each sample was analyzed six times after the first heating and cooling. The variance and standard deviation were calculated using the following formulas:

$$\text{Variance } \mathbf{V} = \text{Sum } (X-Y)^2 / (n-1)$$

Where: X = Delta H obtained  
Y = average of the Delta Hs  
n = number of analyses

$$\text{Standard Deviation } \mathbf{SD} = \text{Square root of Variance } \mathbf{V}$$

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<sup>3</sup> Steven H. Page, Senior Thermal, Elemental Analysis/Autobalance Sales Specialist, *Perkin-Elmer Corporation*. "Thermal Analysis for Textiles: Principles and Applications." Presentation at the AATCC Analytical Methods Workshop, Charlotte, NC, December 9-11, 1998. [Note: Mr. Page is no longer affiliated with *Perkin-Elmer*; his whereabouts are not known.]

<sup>4</sup> E-mail communication from Steven Page to Harold Katcher (subsequently to the author), January 26 1999.

**Table 2. Data obtained for the 100% polyester sample.**

SAMPLE ID/RUN	#DELTA H J/g
10072d.073	39.74
10072e.074	40.76
10072f.075	39.02
10072g.076	39.52
10072h.077	40.70
10072i.078	41.53
Average Delta H:	40.21 J/g
Variance = 0.87	
Standard deviation = 0.94	

**Table 3. Data obtained in the standard deviation study for a sample containing 14/86 polyester/cotton.**

SAMPLE ID/RUN #	DELTA H J/g	% POLYESTER
4700V2.087	5.684	14.54
4700V3.088	5.737	14.68
4700V4.089	6.020	15.40
4700V5.090	5.692	14.57
4700V6.091	5.810	14.87
4700V7.092	5.998	15.35
Variance = 0.023		
Standard Deviation = 0.152		

**Table 4. Data for the standard deviation study obtained for a sample containing 75/25 polyester/cotton.**

SAMPLE ID/RUN NO.	DELTA H J/g	% POLYESTER
10028d.080	27.96	69.53
10028e.081	29.31	72.89
10028f.082	29.61	73.64
10028g.083	30.03	74.68
10028h.084	29.40	73.14
10020i.085	29.52	73.41
Variance = 0.48		
Standard deviation = 0.69		

## Conclusions

There was an agreement between the results obtained by the AATCC Method 20A Chemical Separation procedure and DSC analysis. Where the Chemical Separation tests found more polyester than the stated composition, the DSC results concurred. The findings indicate that DSC is a good and fast procedure that can be applied to the screening of textile samples containing polyester/cotton. A chemical separation procedure (e.g., AATCC 20A) must be used to confirm the DSC data when the results are close to the cut-off limit of 50%, or if there is a change in the classification.

## Recommendations for Further Study

This preliminary study has raised a number of issues recommended for the next phase of research. Those issues include, but are not limited to:

- applying DSC to samples that have first been subjected to size removal;
- the effect of residual natural (cotton) fiber moisture content on the raw data;
- verification that the “artifacts associated with manufacturing” are removed during first heating;
- determination of the heat of fusion for a given polymer, which may be a range of values depending upon the polymer’s nature and processing history; and
- what effect, if any, the polymer morphology (e.g., fibrous vs. particulate) has on the DCS process.

## Citations

1. \_\_\_\_\_. *1998 Technical Manual of American Association of Textile Chemists and Colorists*. AATCC Test Method 20 A-1995—“Fiber Analysis: Quantitative.” AATCC. Research Triangle Park, NC. 1997.
2. \_\_\_\_\_. *1998 Annual Book of ASTM Standards* (Vol. 08.02). ASTM D3417-99 — “Standard Test Method for Enthalpies of Fusion and Crystallization of Polymers by Differential Scanning Calorimetry (DSC).” ASTM. Philadelphia, PA. 1998.
3. Steven H. Page, Senior Thermal, Elemental Analysis/Autobalance Sales Specialist, *Perkin-Elmer Corporation*. “Thermal Analysis for Textiles: Principles and Applications.” Presentation at the AATCC Analytical Methods Workshop, Charlotte, NC, December 9-11, 1998. [Note: Mr. Page is no longer affiliated with *Perkin-Elmer*; his whereabouts are not known.]
4. E-mail communication from Steven H. Page to Harold Katcher, Assistant Laboratory Director, U.S. Customs Laboratory, New York (and subsequently to the author). January 26 1999. [Copy on file].

Sample: NY10305 style 4060 Run DC  
Size: 6.2630 mg  
Method: Polyester/Cotton Blend  
Comment: NY#2-99-10305, style 4060 Polyester, black with prints

DSC

File: C:\TA\Data\DSC\Ny10305d.022  
Operator: LTH  
Run Date: 12-Jan-00 12:15

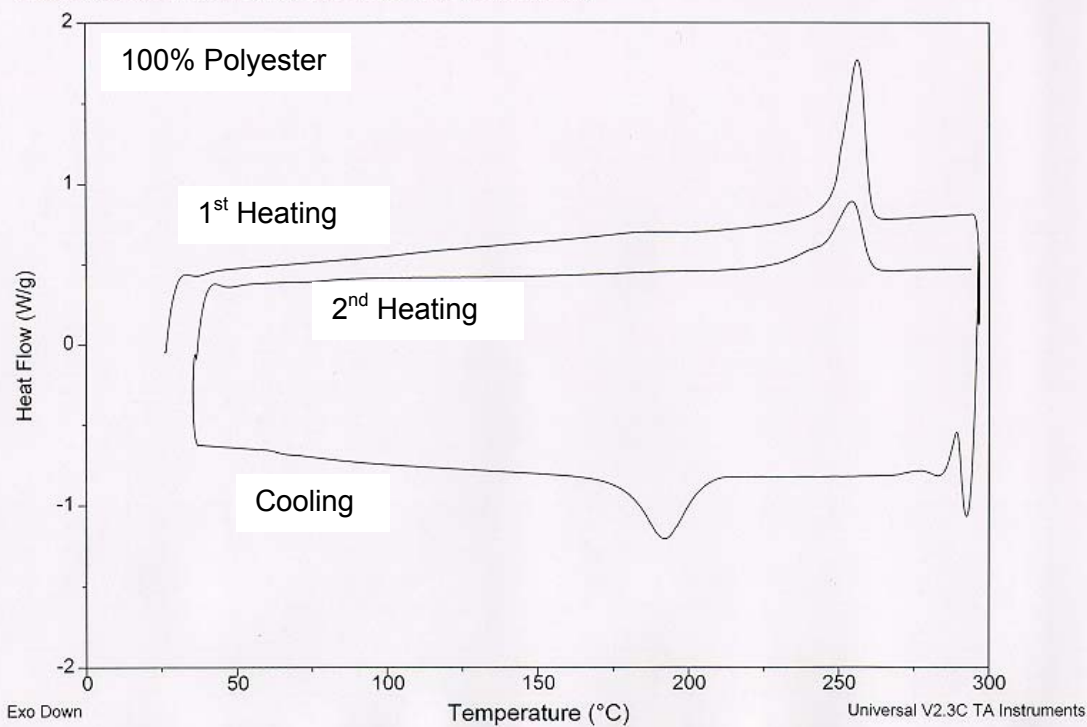


Fig. 1

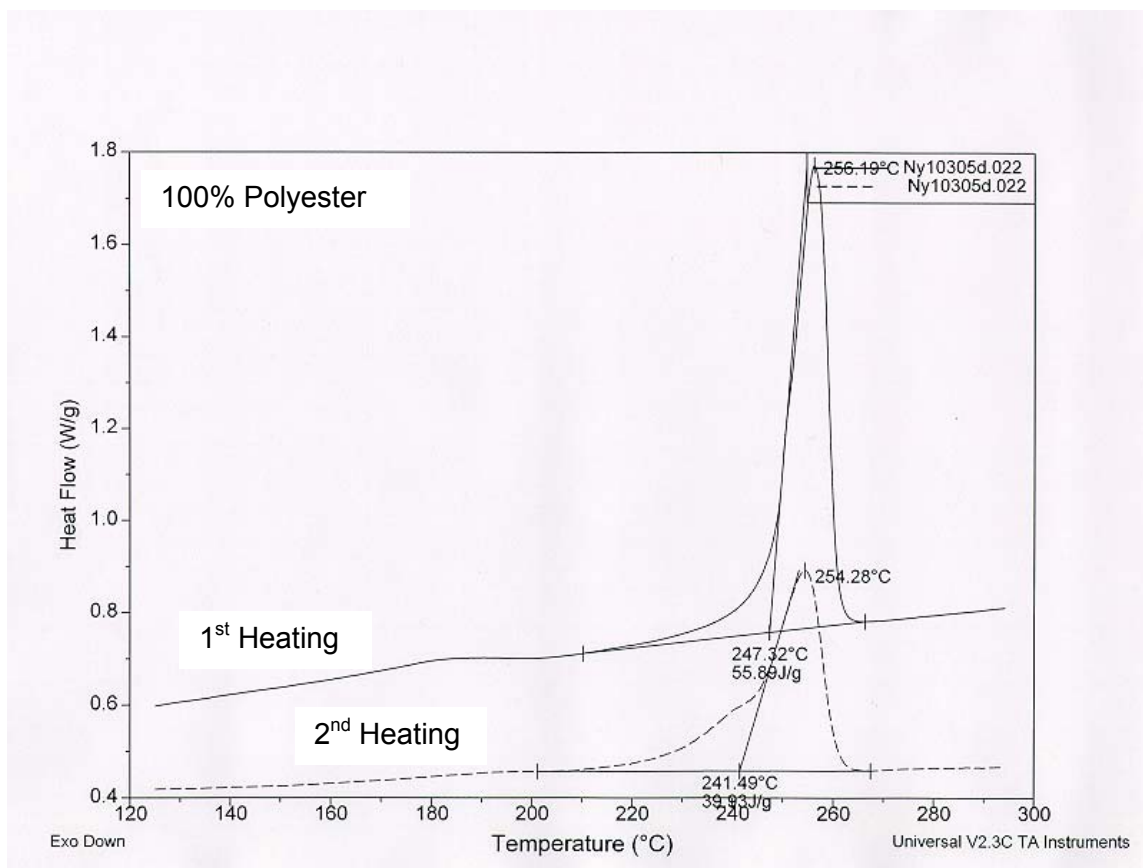


Fig. 2

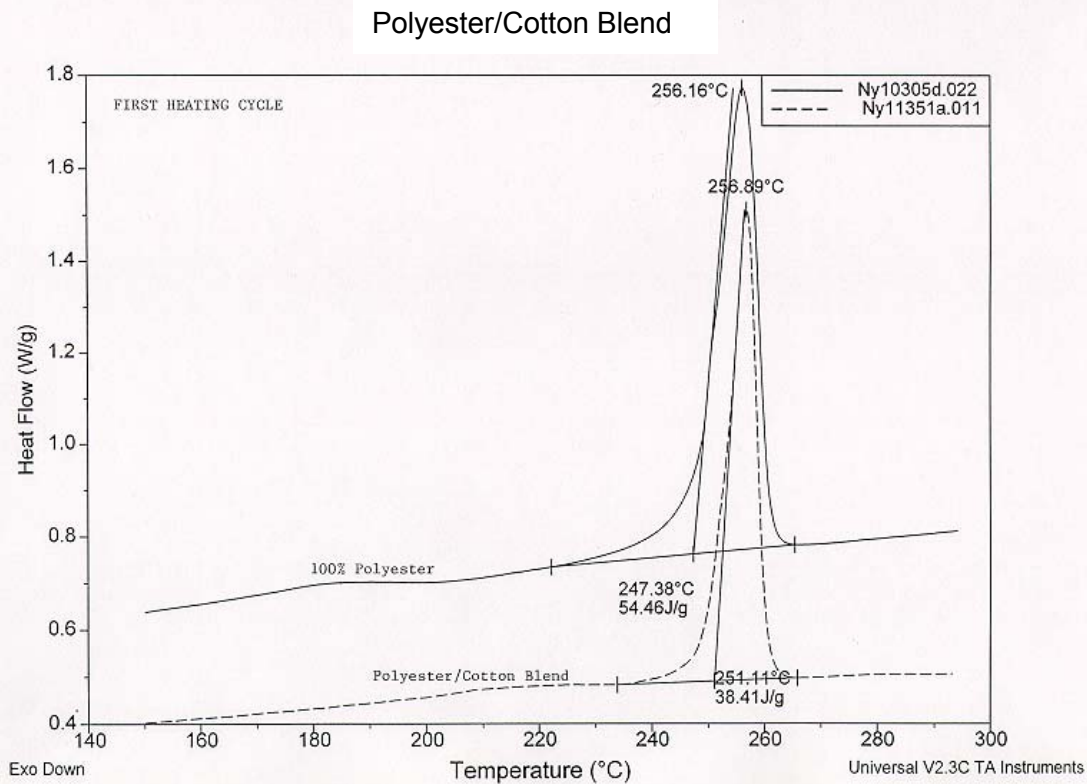


Fig. 3

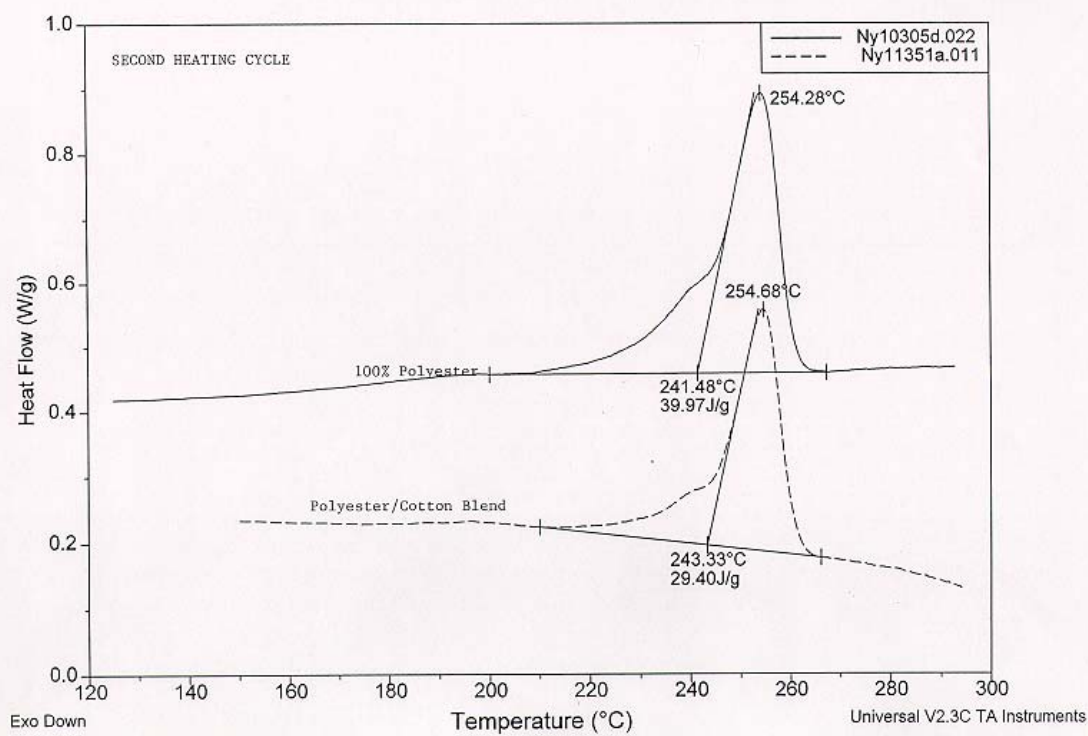


Fig. 4